

Coast to Coast Analytical Specialists

Standard Operating Procedure for Ambient Air Canister Cleaning

Approved By: _____ Approved By: _____
Mary Havlicek, Ph.D. Stephen C. Havlicek, Ph.D.
Chairman of the Board Laboratory Director

1.0 Scope and Application

- 1.1 This SOP describes the cleaning, leak testing and evacuation of SUMMA air sampling canisters at the Garland Laboratory.
- 1.2 Canisters are divided into two groups (ambient and source) based on the anticipated levels of contaminants in the samples. Ambient air canisters are to be used for samples containing contaminants in the parts-per-billion (ppbv) range. Source cans are to be used for samples containing higher concentrations of contaminants (generally $\gg 1$ ppmv). This distinction has virtually disappeared as in-house improvements have been made in the system which allows most source canisters to be cleaned to the TO-14/15 criterion of 0.2 ppbv for target compounds in ambient air. Cans reserved for source samples only (virtually all current samples are source samples, need be cleaned only to 1 PPMV for project-specific target compounds).
- 1.3 This procedure applies to all air sampling canisters used in the Air Toxics Department.
- 1.4 Several separate tasks are outlined in this SOP - Manifold Leak Check (Section 5), Cleaning the Vacuum Trap (Section 6), Cleaning Canisters (Section 5.7-5.9), Analyzing to Confirm Cleanliness (Section 7), Evacuating Cleaned Canisters (Section 8).

2.0 Safety (Wear Safety Glasses or Goggles)

- 2.1 There are several safety considerations unique to the process used in air can cleaning. Air canisters that have been analyzed contain residual sample that may be hazardous if inhaled. Vent cans in a working hood to prevent contact with any residual sample.
- 2.2 Liquid cryogen tanks used in the cleaning process are under high pressure and are very heavy. Use care when transporting tanks. In most cases, these tanks will be on wheels for more convenient transport within the laboratory.

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Dallas Laboratory
2703 National Pl, Garland, TX 75041
Phone (972) 840-8983 Fax (972) 840-8993

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- 2.3 Connecting lines in the cleaning system may whip erratically if disconnected under pressure. Be aware of system pressure before disconnecting any lines.
- 2.4 The liquid used in the cryogenic traps is dangerously cold - burns may result from even momentary epidermal contact.
- 2.5 Canisters are heated during cleaning and become hot to the touch. Use care when handling warm canisters. Allow canisters to cool before submitting for cleanliness checks.

3.0 Equipment and Apparatus - Automated Canister Cleaning System containing

- 3.1 **Vacuum pump** - capable of evacuating sample canister(s) to an absolute pressure of <0.2 in Hg. Oil-less scroll pump is recommended.
- 3.2 **Manifold** - roasted copper manifold with stainless steel connections for simultaneously cleaning several canisters.
- 3.3 **Shut-off valves** - multiple electronically-controlled on-off toggle valves.
- 3.4 **Vacuum gauge** - capable of measuring vacuum in the manifold to an absolute pressure of 0.2 in Hg or less.
- 3.5 **Cryogenic trap** (2 required) - copper open tubular trap cooled with liquid nitrogen to prevent contamination from back diffusion and to provide clean, zero gas to sample canister(s). Both traps may be immersed in the same Dewar flask.
- 3.6 **Pressure gauges** (2) - 0-345 kPa (0-50 psig) to monitor zero air/nitrogen pressure.
- 3.7 **Stainless steel flow control valve** - to regulate flow of zero air/nitrogen into canister(s).
- 3.8 **Humidifier** - pressurizable water bubbler containing organic-free water. Water is replaced weekly, at a minimum, with prepurged organic-free water.
- 3.9 **Blue M oven or equivalent** - capable of fully containing canisters, valve stems and initial stainless steel connectors for heating canisters.
- 3.10 **Dewar containers** - provided for liquid cryogen.

4.0 Preliminary Operations - Normally the system will be running and at the end of a cleaning cycle.

- 4.1 Turn off power to oven

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- 4.2 Check vacuum gauge to determine the status of the cleaning step. A good vacuum indicates the system is in the evacuating step. It is necessary that the system be in this step in order proceed with can removal and initialization. If not in the evacuation stage, wait.
- 4.3 Turn off valve #3
- 4.4 Open Oven CAUTION HOT USE INSULATED GLOVES!
- 4.5 Close canister valves (tighten) STILL HOT!
- 4.6 Push red button on control box to stop the system.
- 4.7 Remove the cleaned canisters. Submit for analysis.

5.0 Leak Checking the Cleaning System Manifold

- 5.1 Verify that sample-containing canisters have been vented in the hood as in 2.1. Place canisters in the oven in the following order: position #2, #1, #3 and #4.
- 5.2 Snug down all fittings with wrench making sure to hold the valve body on each canister with a 13/16 wrench.

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- 5.3 Point all valve knobs towards the center.
 - 5.4 **Hand Tighten the Fittings** - After the nut has been threaded for at least two turns, the 9/16 wrench is used to complete tightening. The body of the valve must be simultaneously held with the 13/16 wrench.
 - 5.5 Close valves #2 & #3, then open valve #1. Apply SNOOP to the now-pressurized system. Look for bubbles. If any bubbles are noted, tighten affected fitting. Verify no more leaks.
 - 5.6 Close valves 1 & 3, then open valve 2. Wait for pressure to stabilize near zero psig.
 - 5.7 Flush canisters.
 - 5.7.1 Close valves 2 & 3, then open valve 1 to fill the canisters with UHP N₂ to about 30 psig. Note pressure of N₂ source is typically higher @ 100-120 psig.
 - 5.7.2 Close valve 1 (valve 3 remains closed), then open valve 2 to vent to near zero psig.
 - 5.7.3 Repeat 3.7.1 and 3.7.2 twice (for a total of 3 times). Finishing pressure should be zero psig.
 - 5.8 Turn on oven. Activate the automatic cleaning system allowing the cans to be alternately exposed to moist zero nitrogen flushing followed by evacuation. Cycling will continue until manually interrupted. Allow a minimum of 4 hr cycling.
 - 5.9 When the last cycle has been completed, evacuate the canisters, close canister valves, turn off oven.
- 6.0 Cleaning the Vacuum Trap
- 6.1 **Empty the liquid nitrogen Dewar.** Allow to come to room temperature. Replace cryogen with warm water (optional).
 - 6.2 Close Valves #6, #7, #8 and #9.
 - 6.3 Open Nitrogen tank valve.
 - 6.4 Increase regulator pressure to 80-100 psig.
 - 6.5 Open valve #8 then open valve 9.
 - 6.6 Allow to vent for a few seconds until all water is gone.
 - 6.7 Close valve #9.
 - 6.8 Turn pressure regulator CCW until it turns easily. The vacuum trap is now cleaned.

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7.0 Analyzing to Confirm Cleanliness

- 7.1 Each cleaned canister is filled with moist zero air and analyzed at 1000 mL sample size to accentuate the presence of low-level target compounds.
- 7.2 The machine report is examined by the analyst. A presumed volume of 250 mL is used for calculation.
- 7.3 Target compounds reported at or above 0.2 ppbv are examined to confirm identity. If identity is confirmed, the canister is sent back for recleaning. Excessive concentrations of non-target compounds may be approved or sent back at the analyst's discretion.
- 7.4 Following the generation of an approved report, but prior to shipping, canisters are pressurized with zero helium to at least 15 psig (record pressure!) for holding until released for shipping. After a minimum of 24 hrs or until needed, canisters remain under pressure. Upon receipt of an order to ship, the canisters are pressure-checked to verify absence of leakage before being connected to the manifold for final evacuation.
- 7.5 **Place new tags on cans.** Record the following on the tag: Batch Number, Date Cleaned, Can Number, pressure into storage.

8.0 Evacuating Cleaned Canisters - Canisters are evacuated because (1) They are delivered evacuated. Canisters are stored under pressure because pressurization allows each canister to be leak checked before delivery while also preventing significant inleakage of atmospheric gases.

- 8.1 Clean vacuum trap.
- 8.2 Open canister valves.
- 8.3 Open Valve #5 only if there is pressure in the system.
- 8.4 Close Valve #5.
- 8.5 Fill Dewar with 2-3 Thermos containers of liquid nitrogen.
- 8.6 Turn vacuum pump on.
- 8.7 Slowly open Valve #7.
- 8.8 Watch vacuum pump gauge. Pressure should fall rapidly.
- 8.9 Open Valve #6. Cans should be evacuated for at least 1 hr.
- 8.10 Close Valve #6.
- 8.11 **Optional:** Close Valve #7 and turn off vacuum pump.

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8.12 Increase pressure on regulator to 5-10 psi.

8.13 Open Valves #1 and #3.

8.14 Remove cans in this order: #4, #3, #1, #2.

8.15 Add post-storage pressure/vacuum to tags on cans.

8.16 File cleaning documentation into files for clean cans. Put cans on shelf labeled "Clean and Ready to Go." Evacuating Clean Cans is done. Also place cleaned cans in proper place, segregating silico cans from non-silico cans and ambient cans from source cans.

9.0 Record Keeping

9.1 Machine reports are filed as hardcopies in the filing area.

9.2 Raw GC/MS data are stored electronically on the server. The server is backed up offsite on a weekly basis using Mozy.

9.3 Just prior to cleaning, old can tags are filed chronologically in a labeled drawer in the can cleaning area.